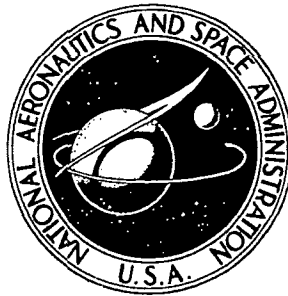


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**DEVELOPMENT AND EVALUATION OF  
VACUUM PRESSURE GAUGE COMPONENTS  
FROM CARBON AND GRAPHITE**

*by David K. Benson and George A. Beitel*

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16. Abstract  <p>A prototype all carbon triode ultrahigh vacuum gage was fabricated and tested. The gage exhibited a sensitivity of <math>3.7 \text{ torr}^{-1}</math> for nitrogen and an x-ray background approximately 0.1 as large as would be expected of a metal gage of the same design. The gage made from these materials, although quite simple and far from optimally designed, did show good sensitivity and durability. A practical technique was developed for bonding carbon components together without metal fasteners. The bond is made with a cross-linked phenolic resin which is converted to vitreous carbon by a careful pyrolysis procedure. The resulting bonds are strong, electrically conductive, and can withstand repeated excursions to 2500 K in vacuum.</p> <p>Measurements of adsorption and outgassing characteristics of four refractory carbons have confirmed that such materials are suitable for use in ultrahigh vacuum and that some are superior refractory metals in many respects.</p> <p style="text-align: center;">18</p>					
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## SUMMARY

The feasibility and advantages of using carbon components in ultra-high vacuum pressure gauges were demonstrated. A prototype, all carbon, triode UHV gauge was fabricated and superficially tested. The gauge exhibited satisfactory sensitivity ( $3.7 \text{ torr}^{-1} (\text{N}_2)$ ) and an x-ray background approximately 1/10 as large as would have been expected of a metal gauge of the same design. A practical technique was developed for bonding carbon components together without metal fasteners. The bond is made with a cross-linked phenolic resin which is converted to vitreous carbon by careful pyrolysis. The resulting bonds are strong, electrically conductive, and can withstand repeated excursions to  $2500^\circ\text{K}$  in vacuum.

Outgassing behavior and adsorption characteristics were measured on four forms of carbon: pyrolytic carbon, Grafoil™ (Union Carbide), vitreous carbon, and pyrolyzed phenolic fiber. The outgassing was measured quantitatively (both amount and composition) in ultra-high vacuum as a function of temperature up to  $\sim 2000^\circ\text{K}$ . The equivalent of several monolayers of gas (mostly  $\text{H}_2$  and  $\text{CO}$ ) are evolved from all of the carbons except vitreous carbon. Less than a monolayer of gas is evolved from vitreous carbon during outgassing to  $2000^\circ\text{K}$ . The adsorption behavior of  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{H}_2$ , and  $\text{CO}$  on the four carbons was studied at low coverages. All of the measured adsorption probabilities were quite low compared to metals (see Table I).

TABLE I

VACUUM CHARACTERISTICS OF CARBONS

Material (source)	Outgassing to 2000°K (monolayers evolved)				Adsorption Probabilities at 300°K			
	<u>H<sub>2</sub></u>	<u>CO</u>	<u>CH<sub>4</sub></u>	<u>C<sub>2</sub>H<sub>6</sub></u>	<u>H<sub>2</sub></u>	<u>CO</u>	<u>O<sub>2</sub></u>	<u>N<sub>2</sub></u>
Vitreous Carbon (Beckwith Carbon Corp., Van Nuys, California)	0.2	0.08	<< 1	<< 1	5 x 10 <sup>-7</sup>	5 x 10 <sup>-4</sup>	~ 3 x 10 <sup>-9</sup>	< 5 x 10 <sup>-7</sup>
Pyrolytic Graphite (Super Temp, Sante Fe, Springs, California)	1.7	0.4	0.25	0.12	5 x 10 <sup>-6</sup>	10 <sup>-5</sup>	--	< 10 <sup>-7</sup>
Grafoil™ (Union Carbide, New York, New York)	20.0	1.2	3.0	2.0	1.5 x 10 <sup>-3</sup>	10 <sup>-2</sup>	--	< 10 <sup>-5</sup>
Pyrolyzed Kynol™ (Carborundum Co., Sanborn, New York)	2.0	10.0	<< 1	<< 1	--	--	--	--

## I. INTRODUCTION

Hot-cathode ionization gauge components and ion sources for mass spectrometers must be made from electrically conductive, refractory materials so that they may be thoroughly outgassed prior to use. If refractory metal components are used, two serious problems arise--desorption and x-ray photoelectron production.

1. Desorption--When a gas molecule strikes a metal surface, the probability that it will be adsorbed is near unity at room temperature. Therefore, even a thoroughly outgassed gauge component or ion source component eventually adsorbs a layer of gas molecules. During ultra-high vacuum (UHV) operation, this reservoir of adsorbed gases may be continuously desorbed thermally or by the impact of stray electrons or photons. This surface supply of gas, at the gauge or ion source, produces an erroneous signal that may be as large or larger than the signal of interest. Since this error signal is affected by past exposure to residual gases, it is known as a "memory effect."

2. X-ray photoelectrons--In many UHV applications electrons are accelerated to a few hundred volts in order to ionize gases. There is a high probability that an energetic electron striking a metal surface will produce a soft x-ray. These x-rays, emitted in all directions, strike other metal surfaces and produce photoelectrons. A photoelectron ejected from an ion collector will appear, in an external circuit, to be identical to an ion striking and being collected at the collector. This error signal, "the x-ray background," establishes a low pressure limit in all simple UHV gauge designs.

Some basic considerations suggest that the memory effect and x-ray background problems might be mitigated if refractory, graphitic materials were used rather than metals. Because of the structure of graphite, we would expect a reduced memory effect. The hexagonal, close-packed basal planes of graphite afford only weak van der Waal's forces for gas adsorption. Only the infrequently exposed, nonbasal planes provide strong bonding sites. The sticking probabilities of residual gases would therefore be expected to be lower on the graphite than on a metal. The x-ray background of graphite is expected to be lower because of the low atomic number of carbon. The intensity of the continuous portion of the x-ray spectrum under electron bombardment is proportional to the atomic number  $Z$  of the target material. Since  $Z = 6$  for carbon, whereas  $Z = 28$  to  $74$  for refractory metals such as nickel or tungsten, the x-ray production could be significantly less for a graphite anode. The probability of an x-ray producing a photoelectron is also a strong function of the atomic number of the target material (ref. 1). The probability of a photoelectric



collision is approximately proportional to  $Z^4$ . Thus, the x-ray background in a UHV gauge could be orders of magnitude lower than in a metal gauge of similar geometry.

These observations suggested that simpler, cheaper UHV gauges could be made from graphitic or carbon materials. The reduced cost, weight, complexity and the concomitant increased reliability would be well suited for many NASA missions. A three-phase, 3-year program was therefore proposed in order to develop and test a superior but simpler carbon UHV gauge. This report summarizes the first year's efforts.

## II. RESULTS

There are two reasons carbon has not been used more often in UHV components--its (undeserved) reputation as a "gassy" material, and problems in its fabrication. Two of the objectives of this project were to dispel the myth that graphite is an inherently gassy material and to develop practical fabrication techniques. It was also necessary to measure sticking probabilities of some gases on graphite for comparison with metals, since many of the pertinent values were unknown. The results of these measurements are described below.

### A. Sticking Coefficients

Adsorption measurements were made with  $H_2$ ,  $CO$ ,  $O_2$  and  $N_2$  on the four carbons. Two problems limited the amount of detailed adsorption data obtainable with  $O_2$  and  $N_2$ . The  $N_2$  sticking coefficients were so low that their measurement taxed the sensitivity of our apparatus. The precision of the  $O_2$  adsorption data was particularly limited because of the memory effect in the mass-spectrometer ionization source. This problem is a good example of the drawbacks of using metal components in UHV ionization sources. The sticking coefficient measurements are summarized in Table I.

The sticking probabilities were measured by repeated flash desorption from filaments of the carbon materials. The sample was mounted in a water-cooled vacuum chamber of volume  $V$  ( $= 3.5 \pm 0.1$  liter), which was pumped at a speed  $S$  (typically  $0.29$  l/s for  $CO$ ) through a conductance limiting orifice. The test gas was constantly leaked into the chamber at a low rate  $L$  (typically  $\sim 0.29 \times 10^{-10}$  torr-l/s). The time dependent behavior of the test gas partial pressure  $p$  is given by the equation

$$V dp/dt = L + \dot{q}(t) - Sp \quad (1)$$

where  $\dot{q}(t)$  is the time rate of gas evolution from the heated carbon filament. Very brief (2-4 sec) filament flashes were used so that the gas evolution time interval  $\Delta t$  was much shorter than the time constant,  $\tau = V/S$ , for pumping the system. Under these conditions, the maximum partial pressure increase is

$$\Delta p_{\max} = \int_0^{\Delta t} \dot{q} dt / V$$

and the amount of gas desorbed,

$$Q = \int_0^{\Delta t} \dot{q} dt = p_{\max} V.$$

If the steady-state partial pressure between filament flashes is  $p$  and the time interval between flashes is  $\Delta T$  then

$$Q = \Delta p_{\max} V = C s p \Delta T \quad (2)$$

where  $s$  is the sticking coefficient and  $C$  is a constant determined by calibration (typically  $19 \text{ torr}^{-1}$  for hydrogen).

The value of the calibration constant  $C$  was determined for each set of measurements by desorbing a saturation (monolayer) coverage of CO from a 0.0025 cm diameter tungsten filament also mounted in the test chamber. In this way a pulse of a known quantity of gas was introduced into the system in just the same way as the gases desorbed from the carbon samples. The maximum increment in the  $\text{CO}^+$  ion current was assumed to correspond to one monolayer ( $5 \times 10^{14}$  molecules/cm<sup>2</sup>) desorbed from the tungsten. The appropriate values of  $C$  for gases other than CO were estimated from the CO calibration by correcting for the known variation of the mass spectrometer sensitivity for the different gases,  $\text{O}_2$ ,  $\text{N}_2$ ,  $\text{H}_2$ .

Figures 1 and 2 are logarithmic plots of typical sets of data for  $\text{H}_2$  and CO adsorption on vitreous carbon. Notice that at very low coverages the slope of the curve is unity as predicted by Eq. (2). The sticking coefficients are estimated from these portions of the data by using Eq. (2). At higher coverages (longer adsorption times) the slope equals 1/2. This square root dependence upon adsorption time  $\Delta T$  (the steady-state partial pressure  $p$  is relatively constant during  $\Delta T$ ) suggests a diffusion mechanism.\* We believe that this portion of the curve represents surface

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\* A characteristic diffusion distance  $\ell$  is given by  $\ell = \sqrt{Dt} \propto \sqrt{t}$  at constant temperature ( $D$  is the diffusion coefficient).

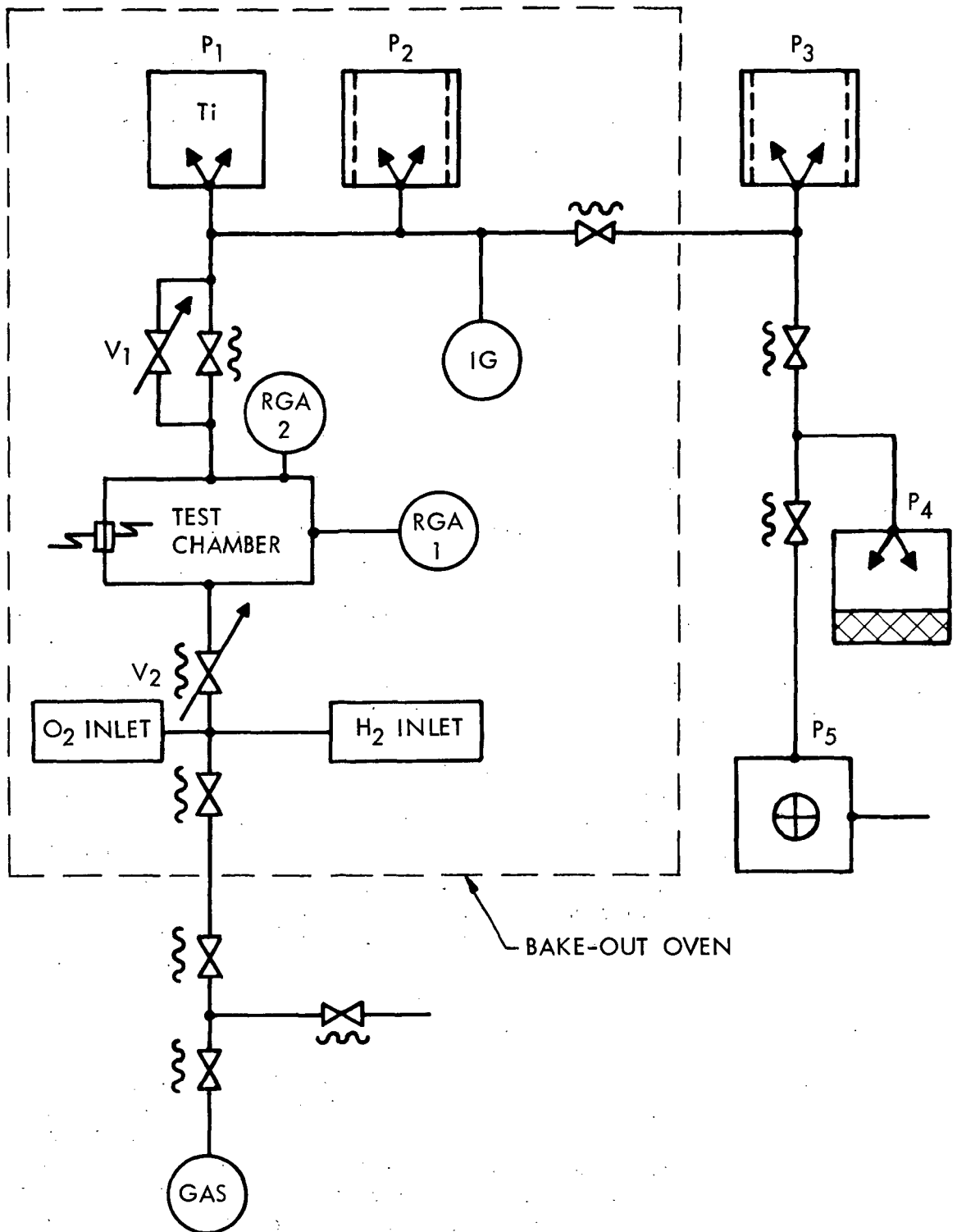


Figure 1 - Schematic Drawing of the Vacuum System.  $V_1$  is a calibrated leak valve and  $V_2$  is an automatic pressure control valve. The  $H_2$  inlet is a nickel diffusion tube and the  $O_2$  inlet is a Vycor diffusion tube. RGA #1 is a magnetic sector mass analyzer and RGA #2 is a rapid response, quadrupole mass analyzer. Construction is stainless steel except for IG,  $H_2$  and  $O_2$  inlets. Standard AVS symbols are used.

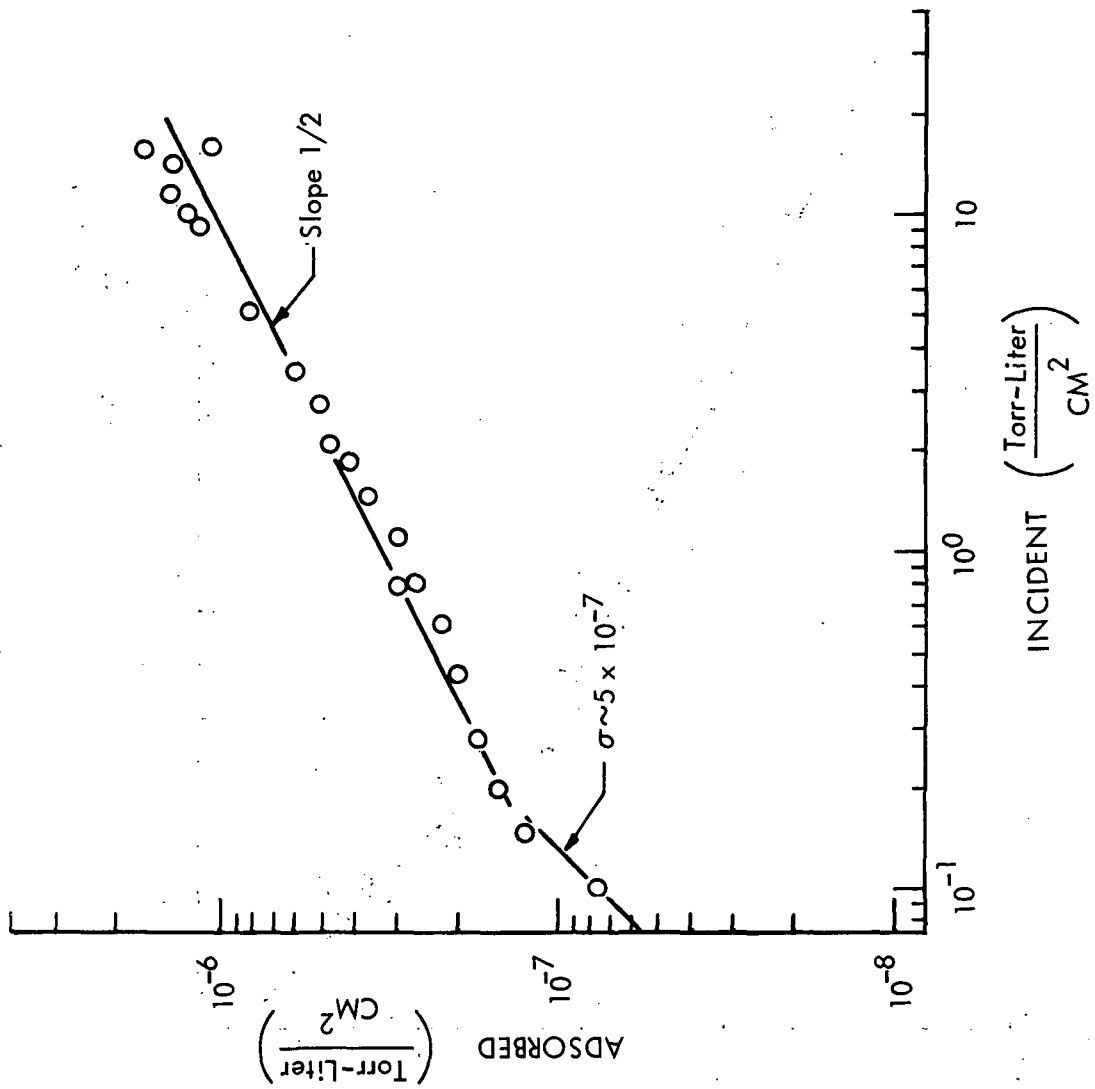


Figure 2 - Adsorption of Hydrogen on Vitreous Carbon at 300°K

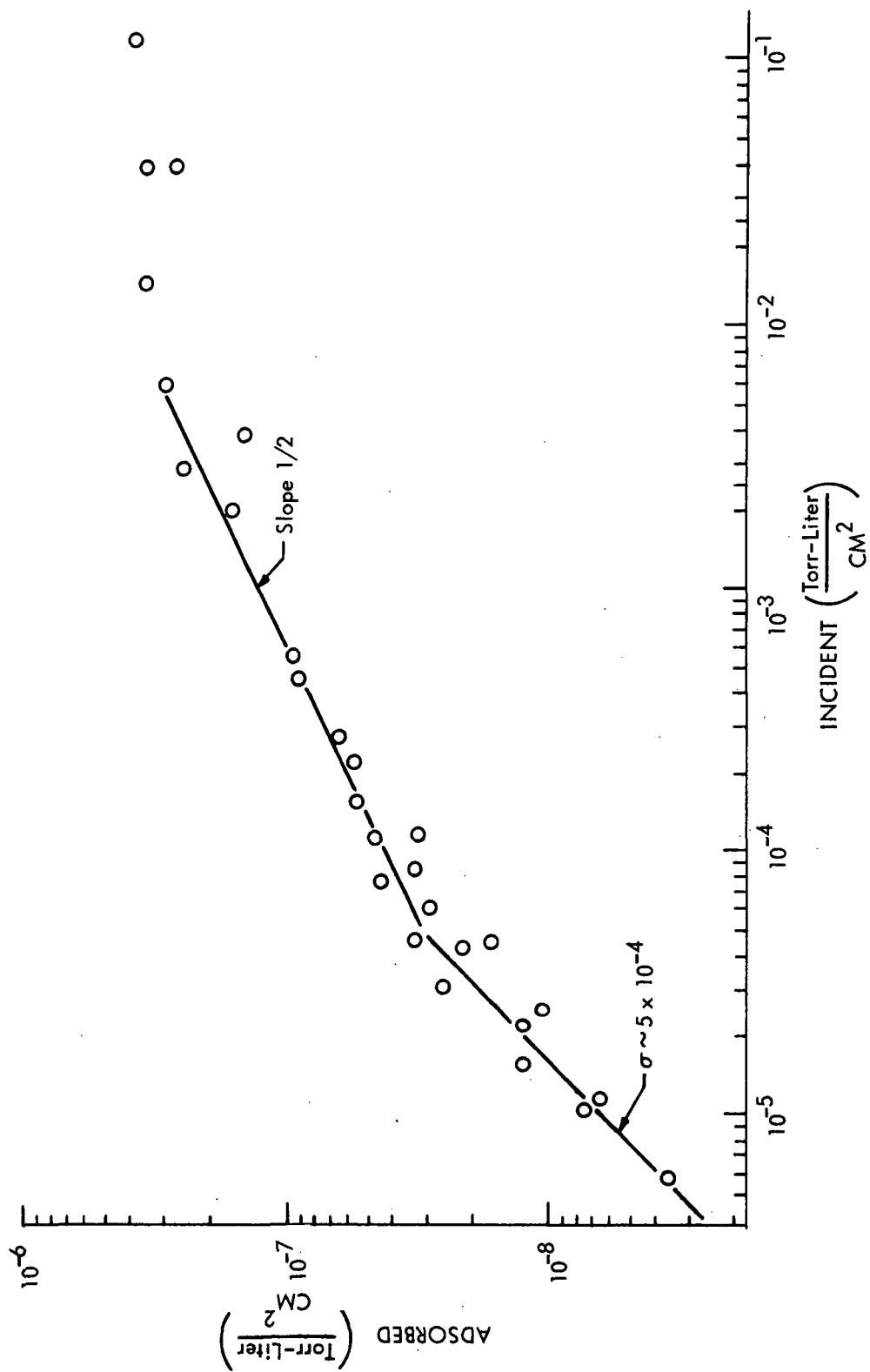


Figure 3 - Adsorption of Carbon Monoxide on Vitreous Carbon at 300°K

migration of adsorbed gas from the clamped portion of the sample. These portions of the samples are never completely degassed and therefore represent reservoirs from which adsorbed gas can migrate during long intervals.

Our attempts to measure the sticking coefficients of  $N_2$  on pyrolytic graphite illustrate the problems of our measurements with this gas and with oxygen. Even when the steady-state partial pressure of  $N_2$  was raised to 1,000 times the background CO partial pressure ( $\sim 10^{-10}$  torr), all of the desorbed gas appearing at  $m/e = 14$  could be accounted for as doubly ionized CO. Perhaps no nitrogen is adsorbed. However, the value quoted in Table I is based on an upper limit in which we assume that all of the  $m/e = 14$  signal is due to desorbed nitrogen under these conditions.

It was not possible to estimate an upper limit for the room temperature sticking probability of  $O_2$  on the carbons as we did with  $N_2$ . When the  $O_2$  partial pressure was increased well above the residual CO pressure, CO produced by the reaction of  $O_2$  with the heated sample could not be distinguished from the desorbed gas which was also evolved as CO. It was possible, however, to measure the reaction rate of  $O_2$  as a function of sample temperature and extrapolate the rate to room temperature. Both steps were taken in the case of vitreous carbon only. The sticking coefficient of  $O_2$  on vitreous carbon quoted in Table I is such an estimate, based on the oxidation rate data shown in Figure 3.

## B. Outgassing

Mass-spectra analyses were made of gases desorbed from carbon samples at increasing temperatures up to about 2000°K. Samples were initially outgassed above 2000°K, exposed to laboratory air for several hours at room temperature and then tested in UHV  $\sim 10^{-10}$  torr, following a normal 24-hr bake at 300°C. A quadrupole mass analyzer (Ultek Model 200) was used along with a multichannel oscillographic recorder. Mass spectral scans from  $m/e = 1$  through 50 were made once per second before, during, and for a few seconds after each brief (4 sec) period of flash heating. The samples were heated (by electrical conduction) in increments of about 100°K up to 2000°K. Between flashes the sample was allowed to cool to room temperature as the background pressure was returned to the  $10^{-10}$  -  $10^{-9}$  torr range. During each flash the pumping speed of the 5-liter system was reduced to 0.08 liters/sec (nitrogen equivalent). The system pumping speed time constant was 60 sec. Since the flash duration was only 4 sec and the sampling interval 1 sec, the quantity of gas evolved  $q_i$  could be adequately represented by

$$q_i = \Delta p_i V = C_i^m \Delta T_i^m V \quad (3)$$

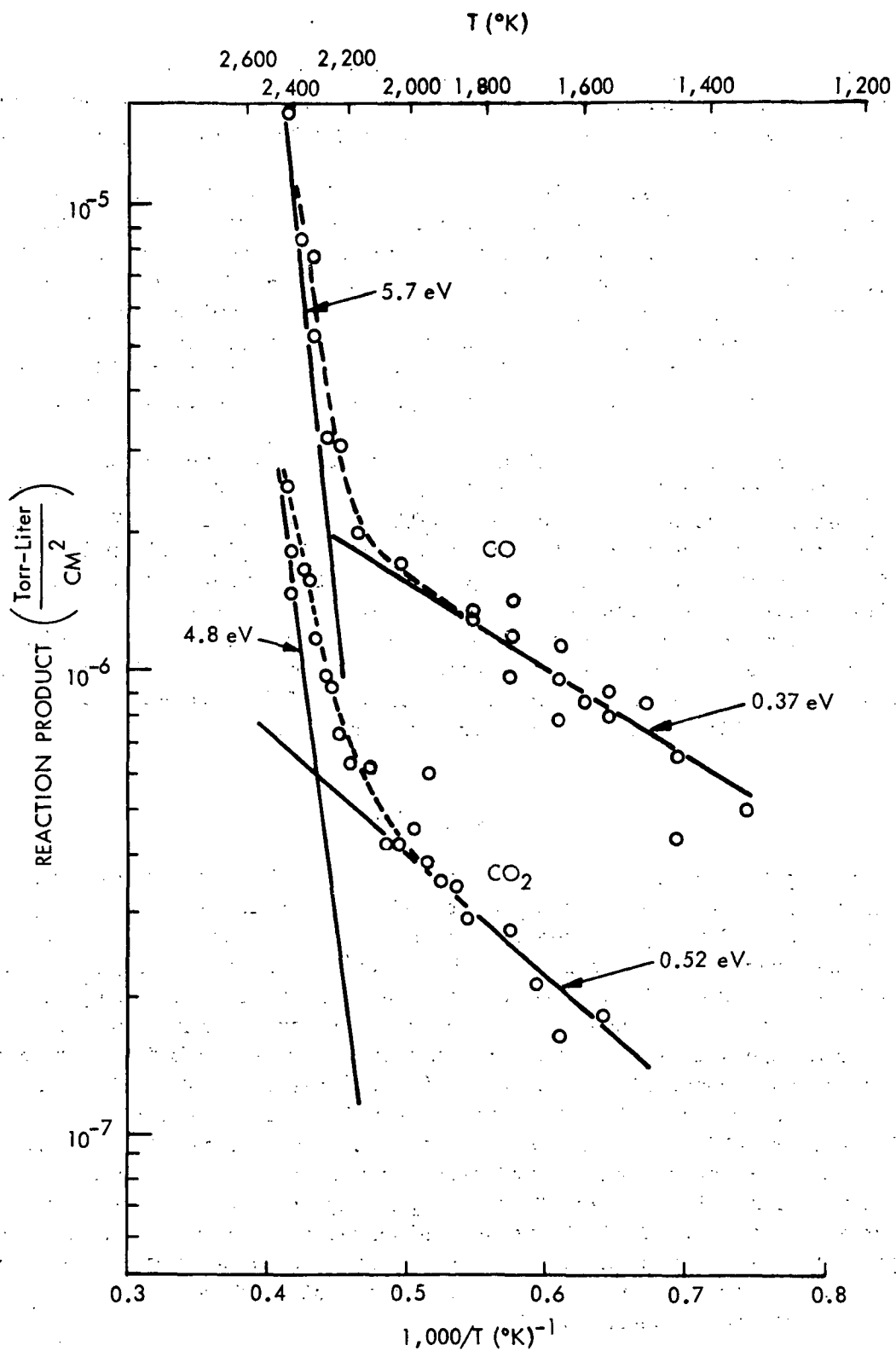


Figure 4 - Oxidation of Vitreous Carbon at  $5.6 \times 10^{-5}$  Torr Partial Pressure of Oxygen

where  $\Delta p_i$  is the increase in partial pressure,  $V$  is the system volume,  $\Delta I_i^m$  is the change in the ion current and  $C_i^m$  is a calibration constant ( $i$  refers to the  $i$ th gas and  $m$  refers to a particular mass peak in the spectrum of the  $i$ th gas).

A 0.00254 cm diameter tungsten filament with a surface area of 0.048 cm<sup>2</sup>, mounted within the test chamber, was used to calibrate the mass spectrometer, as mentioned in the previous section. During each run, the tungsten filament was thoroughly degassed and allowed to adsorb CO to saturation. The tungsten filament was then flashed,  $\Delta I_{CO}^{28}$  was noted, and assumed to correspond to one monolayer.\* This allowed us to obtain a standardized value for  $C_{CO}^{28}$  for each test run and we could estimate the other  $C$ 's on the basis of published ionization cross sections. All gases except H<sub>2</sub> were calibrated in this fashion. Because of the peculiar construction of our vacuum system, we were able to back-fill the entire system with 99% pure H<sub>2</sub> by heating the sublimators. It was then a simple matter to calibrate the mass spectrometer for  $m/e = 2$  against a precalibrated Bayard-Alpert gauge, located on the sublimator side of the pumping orifice.

Sample temperatures during outgassing were inferred from temperature versus conduction current calibration measurements made on each sample in an auxiliary vacuum system. A Pyro Photomatic recording pyrometer model A-120 (Pyrometer Instrument Co., Northvale, New Jersey) was used. The thermal time constants were measured for typical samples in order to select a minimum outgassing flash duration which still allowed thermal equilibrium to be reached at the lowest flash temperatures. As expected, the time constant  $\tau$  was approximately proportional to the flash temperature  $T$  raised to the negative fourth power, i.e.,  $\tau \propto T^{-4}$ . The longest time constant was well under 1 sec.

It became necessary to measure the emissivity of vitreous carbon during these calibration experiments as no value could be found in the literature. A 2 mil diameter chromel-alumel thermocouple was imbedded in a slotted vitreous carbon bar and bonded in place with phenolic resin which filled the slot. The resin was then pyrolyzed to vitreous carbon. The emissivity was calculated by comparing the sample temperature as indicated by the thermocouple with the temperature as indicated by the pyrometer aimed at the opposite side. No correction was made for the effects of the intervening glass window. The value of the emissivity averaged over the temperature range 1100°-1500°K was  $0.89 \pm 0.05$ .

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\* One monolayer =  $5 \times 10^{14}$  molecules/cm<sup>2</sup>.



All degassing data are presented graphically (Figures 4 through 7) as the cumulative gas evolved in units of monolayer equivalents normalized to the gross geometrical surface area of the test sample versus the temperature. Each curve shows a temperature threshold for gas evolution, 950°-1000°K, which is about 300°K higher than the bake-out temperature. In the case of CH<sub>4</sub> and C<sub>2</sub>H<sub>4</sub>, there is a definite temperature at which the outgassing rate goes through a maximum. Both H<sub>2</sub> and CO outgassing rates apparently continue to increase above 2000°K. However, the gas which appears at temperatures above 2000°K has been identified as desorption from the chamber walls and sample fixtures caused by low energy electron bombardment. The electrons are emitted thermionically from the sample and accelerated to the walls or filament leads by the voltage drop (~ 20-30 V) occurring across the conduction heated sample. Effects of sample polarity, AC versus DC current and estimates of electron desorption rates are all consistent with this interpretation of the high temperature data.

The pyrolytic graphite, Grafoil, and vitreous carbon samples were in the shape of ribbons with well-defined dimensions. Surface areas were calculated from these dimensions. The pyrolyzed Kynol fibers were in a loose yarn. In order to estimate a surface area for this sample, the bundle of fibers was potted in plastic, sectioned, and photomicrographed at 100 X magnification. From the micrograph, it was estimated that the average fiber diameter was 7 μm (in agreement with manufacturers' specifications) and that there were 575 fibers/bundle. Therefore, the estimated surface area of the test sample (1.2 in. in length) was 3.86 cm<sup>2</sup>.

As expected, the major gases evolved are H<sub>2</sub> and CO. The Grafoil, with its loosely packed lamellar structure, had the highest gas content and the most hydrocarbons. Vitreous carbon was the cleanest carbon with only about 0.01 times as much gas as the Grafoil.

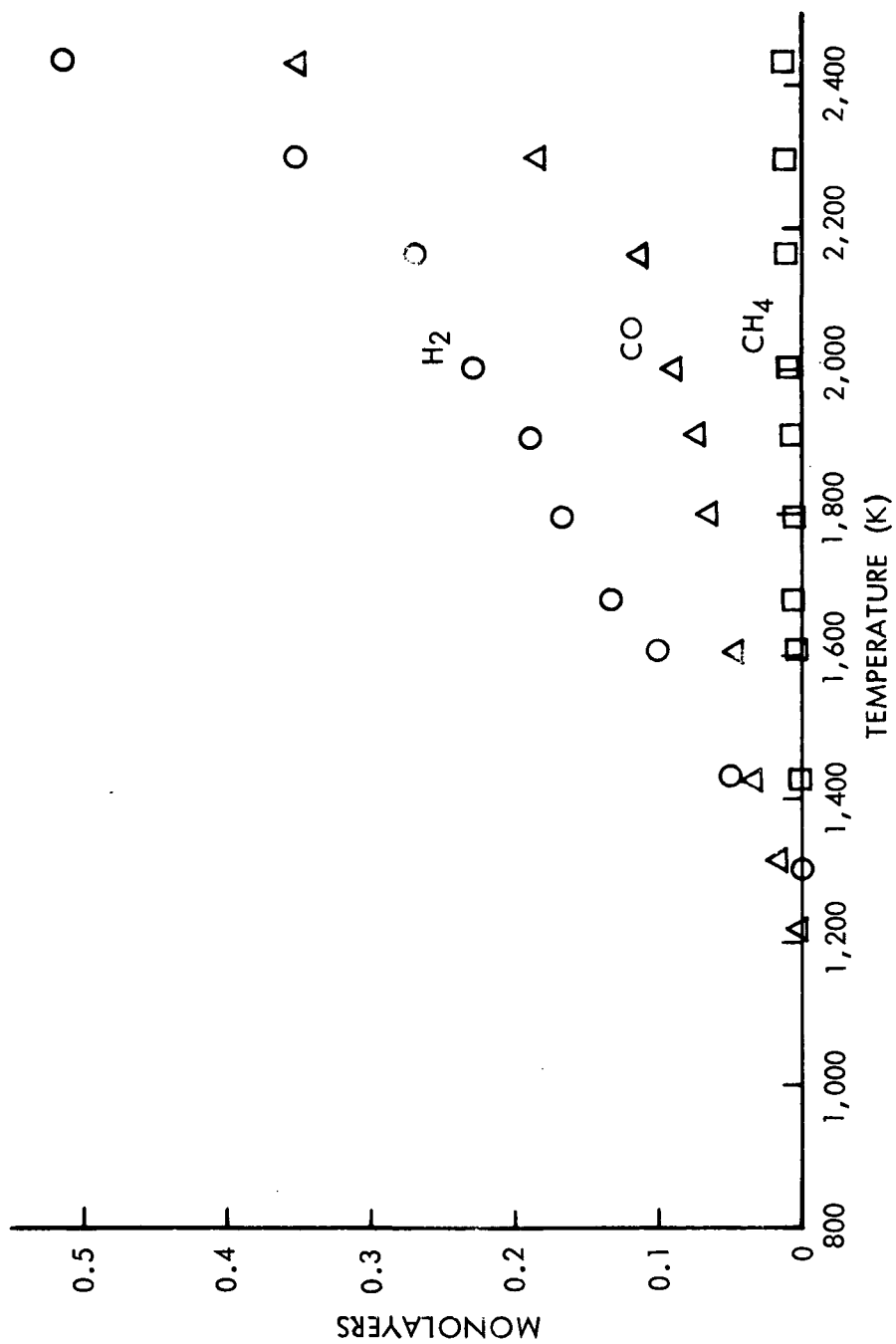


Figure 5 - Outgassing From Vitreous Carbon

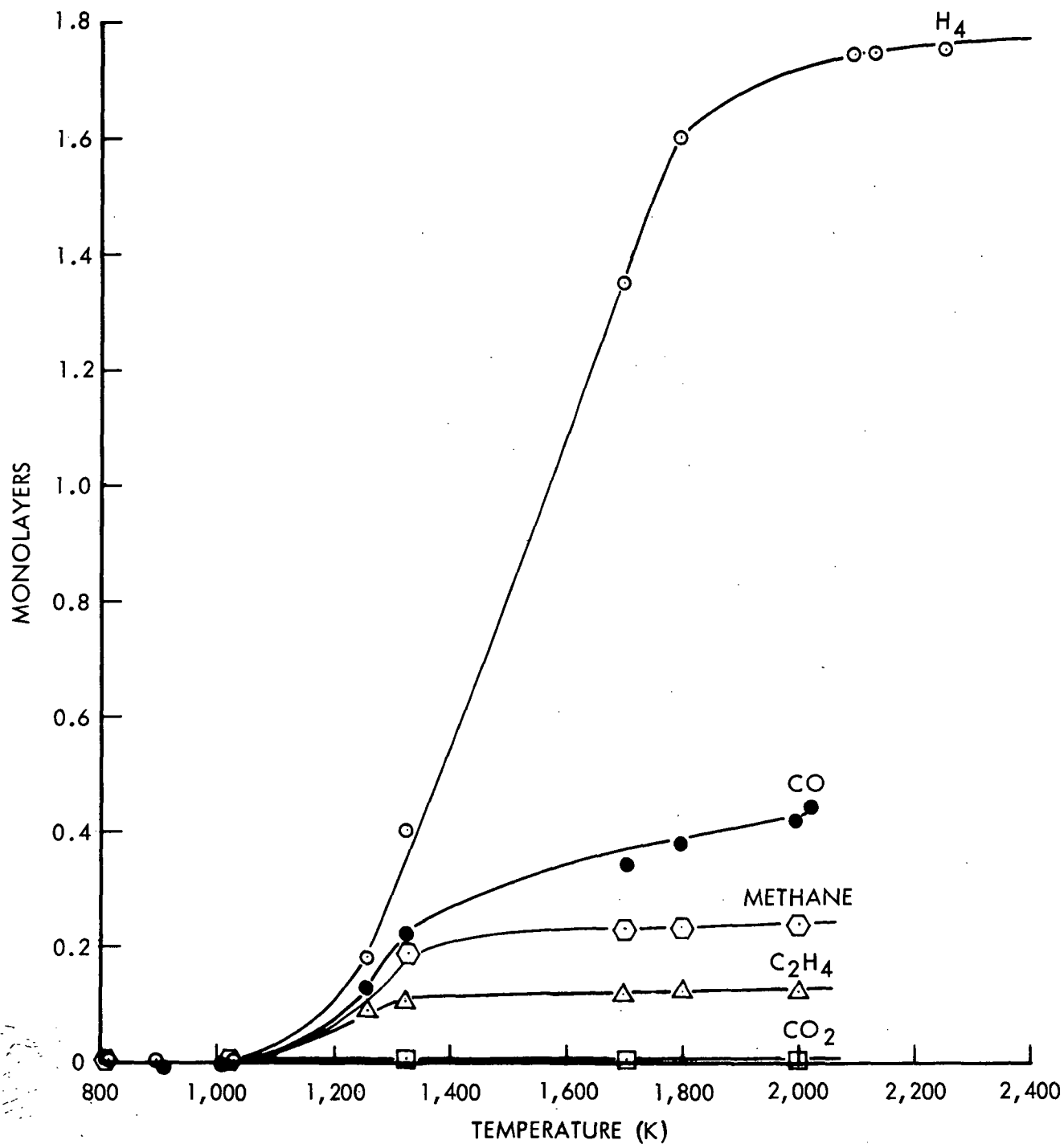


Figure 6 - Outgassing From Pyrolytic Graphite

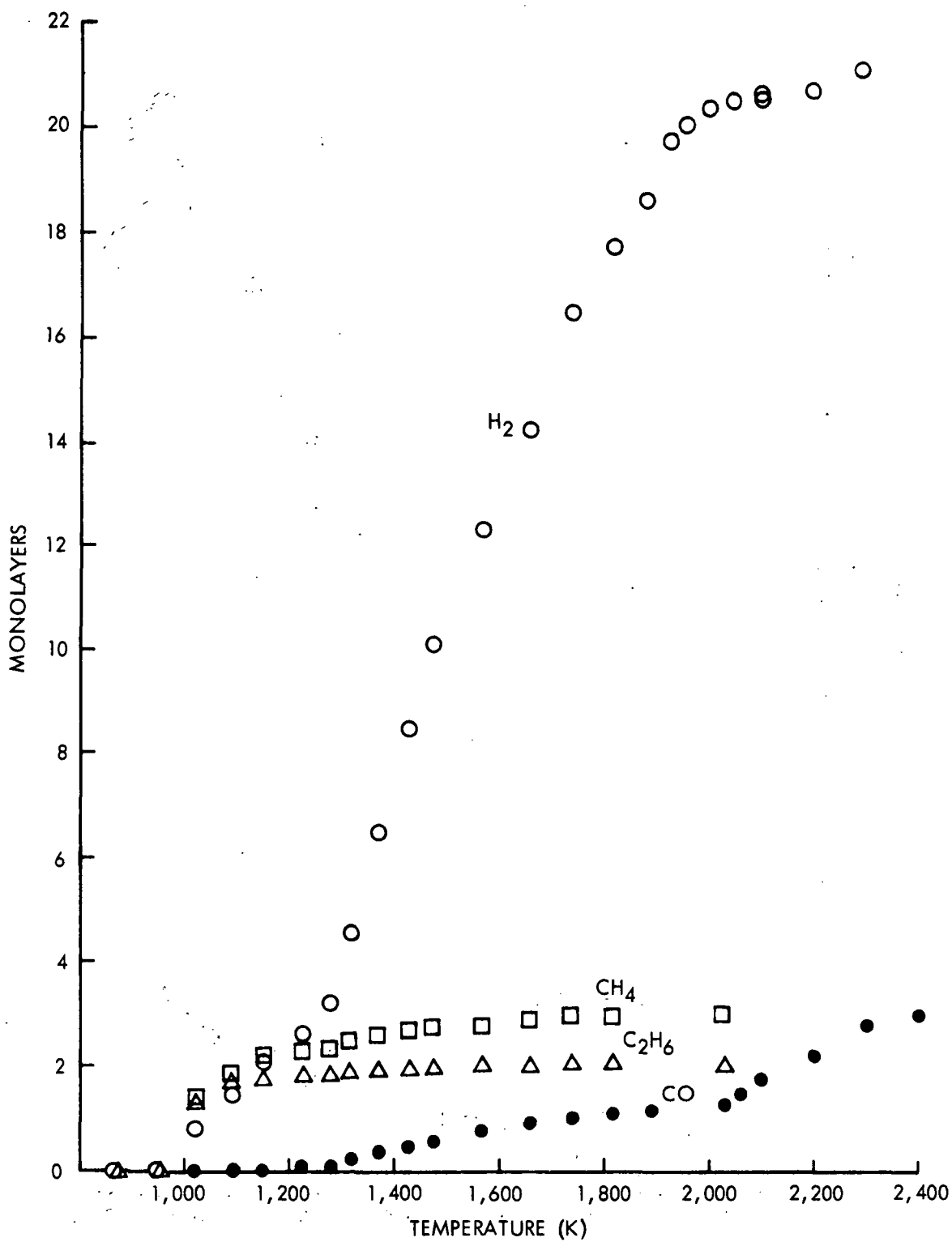


Figure 7 - Outgassing From Grafoil™

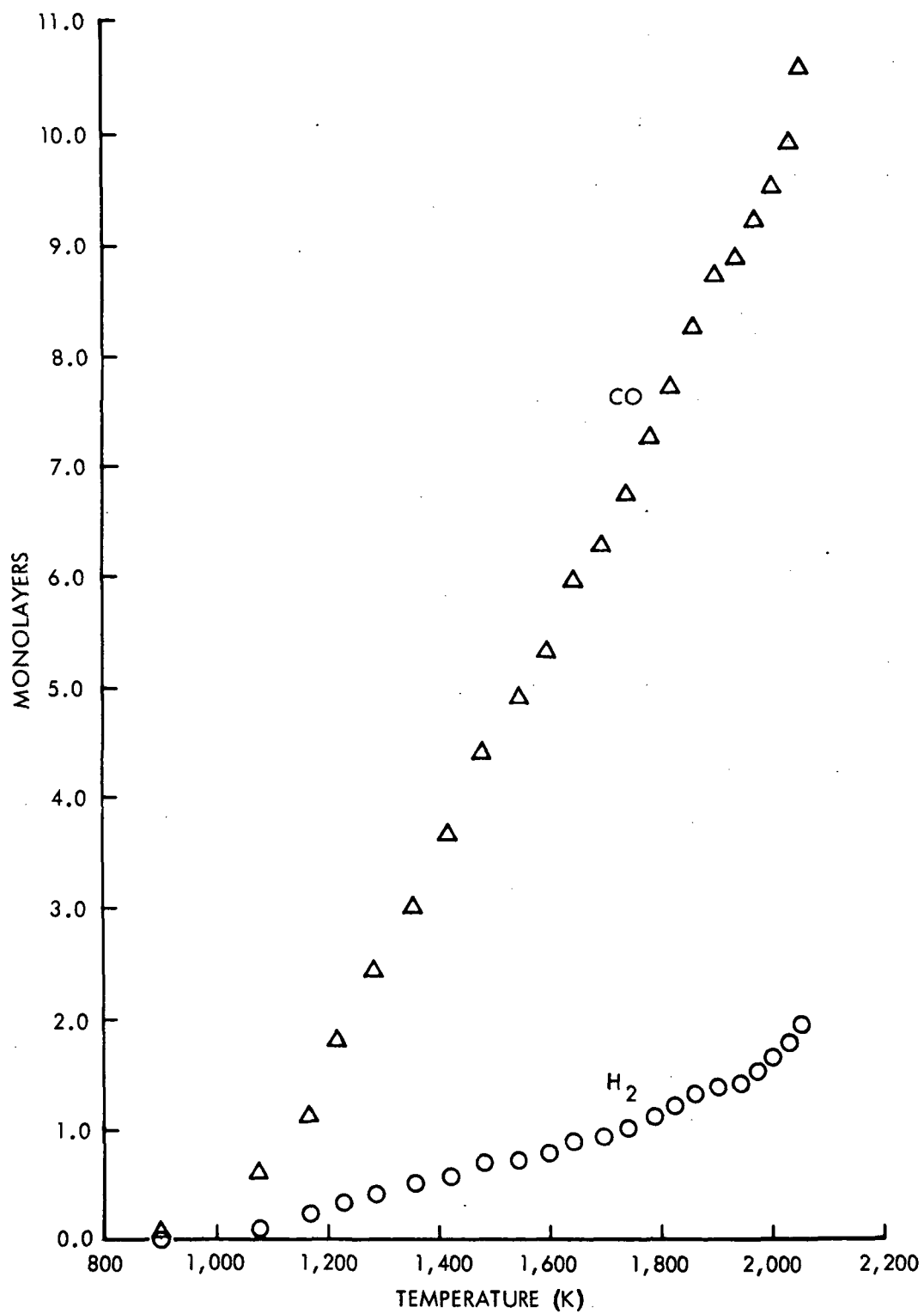


Figure 8 - Outgassing From Pyrolyzed Phenolic Fiber Yarn (Kynol™)

### C. Gauge Fabrication

Conventional techniques for fabricating metal components such as welding or brazing cannot be used on carbon. If metal parts are to be completely excluded from the construction, then screws, bolts, and rivets are also impractical. How, then, is a complex, small device such as a vacuum gauge to be fabricated so that it can withstand repeated outgassing at temperatures as high as 2500°K?

We have developed a practical answer to this question. We have found that carbon parts can be bonded together with a phenolic resin which can subsequently be converted to vitreous carbon by a careful pyrolysis procedure. The resulting bonds are strong (although somewhat brittle), electrically conductive, and capable of withstanding repeated excursions to 2500°K in vacuum. A further advantage of the vitreous carbon is its excellent outgassing and adsorption behavior described in Section II.

The vitreous carbon bonds were made by a sequence of steps: first, a mixture of resorcinol plus 5% formaldehyde is applied to the carbon surfaces and they are clamped together. The resorcinol is converted to phenolic resin by a condensation reaction in an air furnace at ~ 110°C. After several hours at this temperature (or a correspondingly shorter time at a higher temperature--up to about 250°C) the bond is rigid and fairly strong. The clamps can be removed and the pyrolysis accomplished by slowly heating in an inert gas (~ 3°/min) to 750°C and then more rapidly to 1000°C. After the 1000°C pyrolysis, the bond is ready for vacuum use; however, a final outgassing at ~ 2200°C in an auxiliary vacuum system is advisable.

Fabricating delicate parts, such as a grid for a triode vacuum gauge, from carbon is another serious problem. Two satisfactory designs were fabricated. One consisted of a perforated cylinder rolled from 0.005 in. thick Grafoil tape and bonded to pyrolytic graphite supports. The perforations were made with a SiC abrasive powder entrained in an air stream.\* A copper screen was used as a mask. With this technique the maximum practical grid transparency is about 50%. A 90% open area would be desirable.

A more open grid (~ 90% open) was fabricated from Kynol fibers impregnated with a phenolic resin and finally converted to vitreous carbon. Although this structure was acceptable, the fine vitreous carbon coils were extremely fragile.

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\* An S.S. White Airbrasive Unit, Model F was used.

We have constructed four prototype graphite gauges by the techniques described above. The first two included tungsten filaments and were used to test the integrity of the grid and collector. The third gauge was built entirely of carbon materials and its design and performance is described in detail below and in Table II. Graphite Gauge No. 4 was built by means of our best available techniques (with the exception of not using a vitreous carbon grid). It is shown in Figure 8. The performance of Gauge No. 4 has not been studied.

The parameters of interest for Graphite Gauge No. 3 (GG3) have been collected and are presented in Table II. This gauge had the normal triode configuration, and is nearly identical in structure to the gauge depicted in Figure 9.

TABLE II  
CONSTRUCTION AND PERFORMANCE CHARACTERISTICS OF  
PROTOTYPE CARBON TRIODE GAUGE NO. 3

		<u>Comments</u>
Gauge type	Normal triode	See Figure 8
Materials:		
Filament	Pyrolytic graphite	Parts attached with phenolic resin cured to vitreous carbon.
Grid	Grafoil	
Collector	Grafoil	
Supports	Pyrolytic graphite	
Dimensions:		
Overall length	2 cm	50 cm <sup>2</sup> surface area
Grid diameter	1 cm	
Collector diameter	1.5 cm	
Grid transparency	55%	
Filament:		
Power	35-45 W	Maximum temp = 2400°K
Emission	0.1-10 mA	
Degassing power	500 W	T > 1800°K
Gauge sensitivity	3.7 torr <sup>-1</sup> (N <sub>2</sub> )	= I <sup>+</sup> /(I <sup>-</sup> P)
X-ray current:		
V <sub>g</sub> = 1000 V; I <sup>-</sup> = 1 mA	6 x 10 <sup>-11</sup> A	± 10%
V <sub>g</sub> = 100 V; I <sup>-</sup> = 0.1 mA	2 x 10 <sup>-12</sup> A	estimated
X-ray limit	5.5 x 10 <sup>-9</sup> torr (N <sub>2</sub> )	I <sub>x-ray</sub> <sup>+</sup> = I <sub>gas</sub> <sup>+</sup>

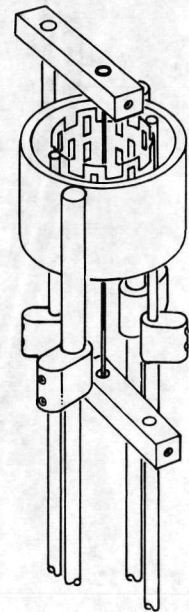
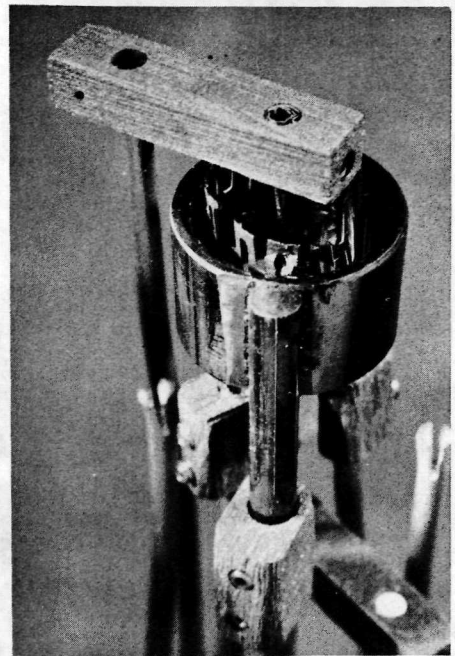
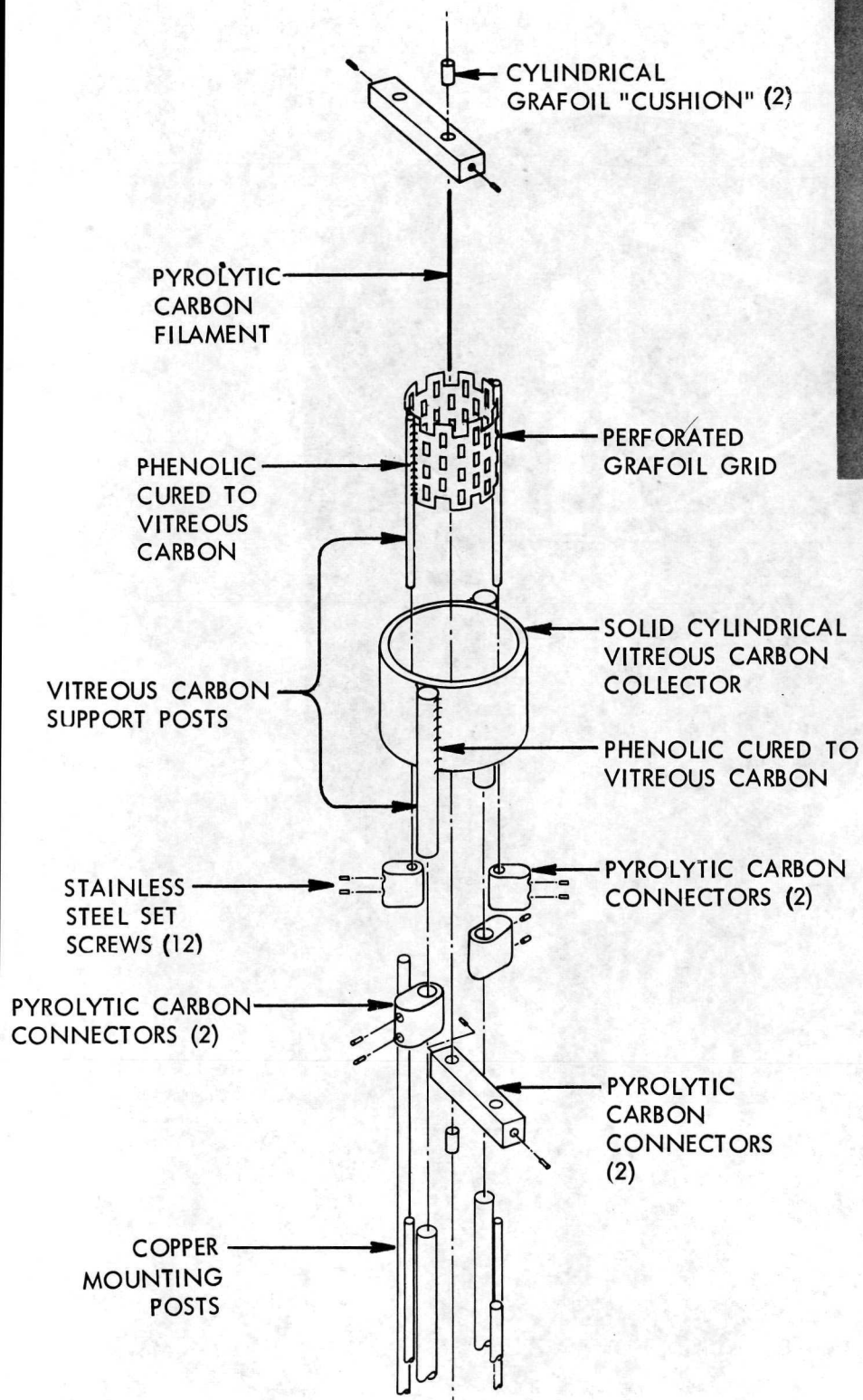


Figure 9 - Prototype Carbon Vacuum Gauge No. 4



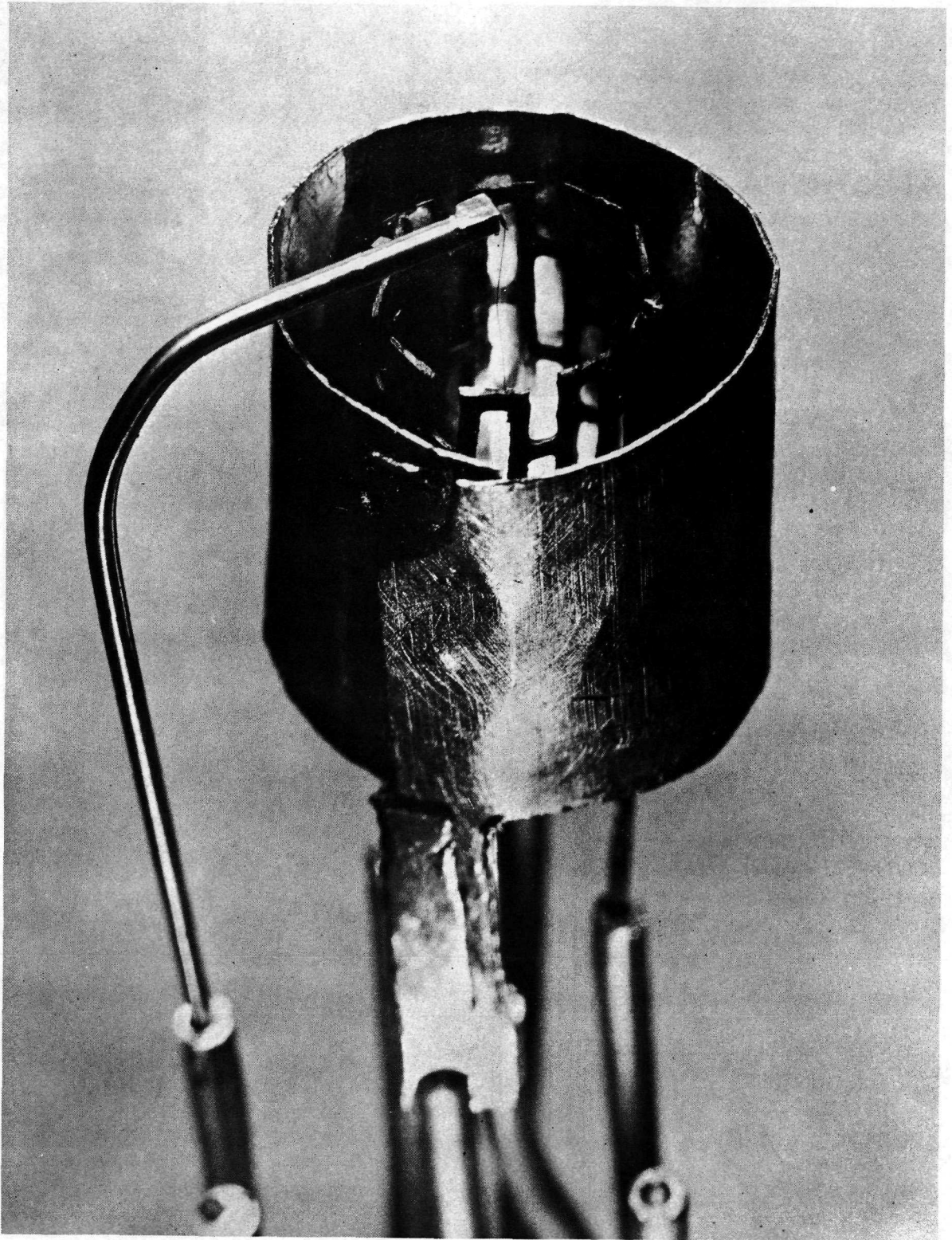


Figure 10 - Prototype Carbon Vacuum Gauge No. 2

#### D. Gauge Testing

Each gauge component was held by two supports and could be heated by conduction current. The gauge could be degassed with a maximum of 500 W heating power (27.5 A at 18 volts) reaching a collector temperature of 1800°K, while the enclosed grid reached an estimated 2000°K.

The filament required 45 W to reach 2400°K at which temperature it could supply 20 mA emission current. The gauge was normally operated at 0.3 mA which required 37 W (5.5 A at 6.7 V) heating power.

After the normal system bake-out (300°C for 24 hr) the system pressure fell below  $10^{-10}$  torr indicating that the graphite gauge at room temperature had a nonmeasurable outgassing rate. When operated as a gauge (0.3 mA emission, 150 V filament-to-grid, 37 W filament power) the gauge (collector, grid, and supports) had an outgassing rate of about  $5 \times 10^{-9}$  torr l/cm<sup>2</sup>. It is expected that the 37 W would raise the temperature of the grid and collector to about 750°K, 130°K above the bake-out temperature. Considering the bulk of graphite in this gauge and the outgassing data we have collected on the various graphites, high temperature degassing is obviously necessary to measure pressures below  $10^{-8}$  torr.

After the gauge had been thoroughly degassed, it was calibrated against a Bayard-Alpert gauge in the pressure range  $10^{-8}$  to  $10^{-6}$  torr. The gauge had a sensitivity of 3.7 torr<sup>-1</sup> (N<sub>2</sub>) which compares favorably with the expected value:

$$P = I^+ / (I^- P) = \sigma_{N_2} \bar{\ell} n I^- / (I^- P) = \sigma_{N_2} \bar{\ell} n / P = 5 \text{ torr}^{-1}$$

where  $I^+$  = ion current;

$I^-$  = electron emission current;

$\sigma_{N_2}$  = ionization cross section;

$n$  = the number of molecules/cm<sup>3</sup>;

$\bar{\ell}$  = mean path length of electrons ( $\sim 0.5$  cm, the radius of the grid).

Although GG3 was not mounted in a chamber with a high enough pumping speed to insure that the ambient pressure was much less than the x-ray limit, preliminary x-ray background data were obtained. Collector current was measured as a function of filament-to-grid voltage ( $V_g$ ). The result is shown in Figure 10. The collector current for  $V_g > 200$  V behaves like

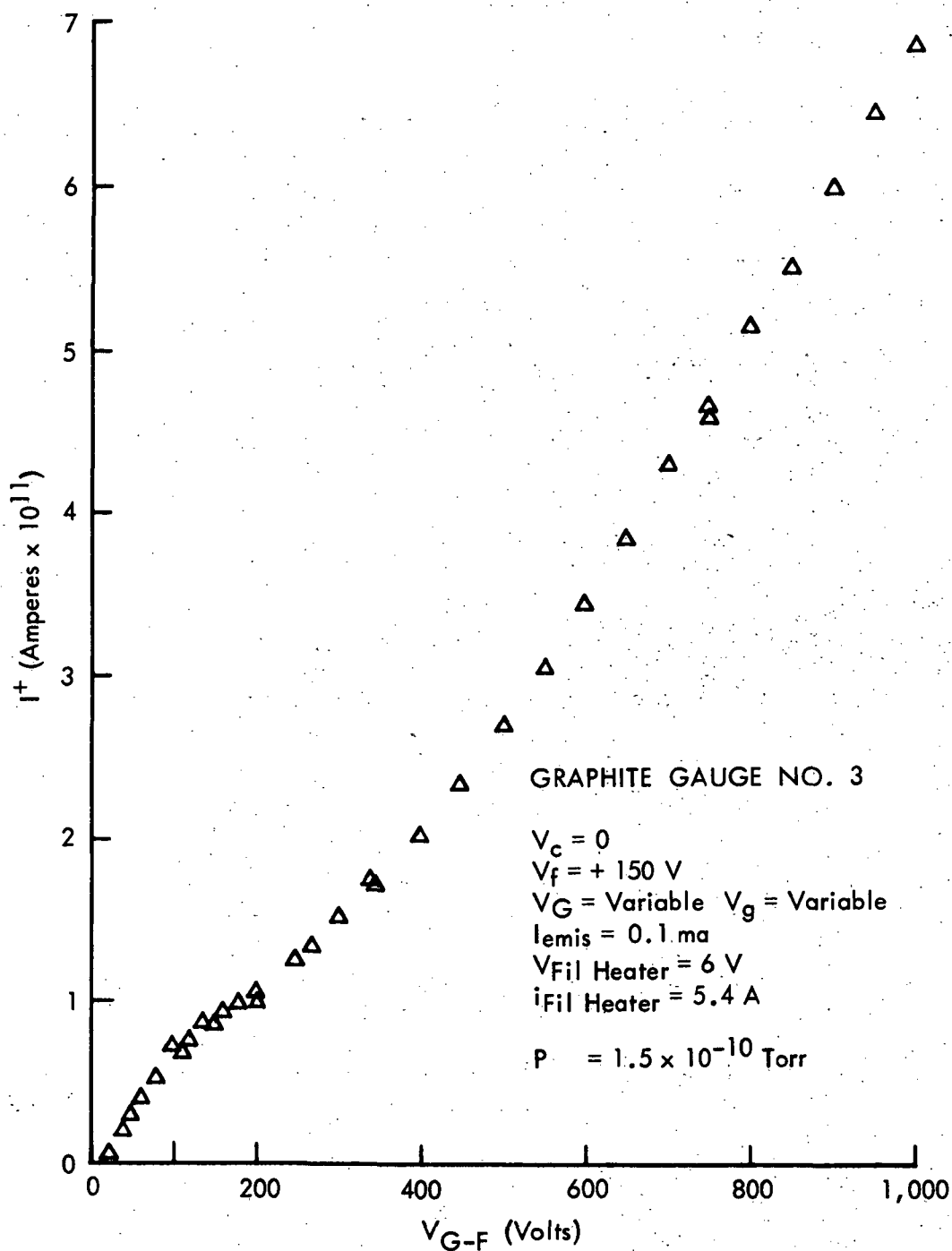


Figure 11 - Ion Current  $I^+$  and X-ray Background  $I_x$  for Prototype Carbon Gauge No. 3

x-ray background. If it is assumed that  $I_{x\text{-ray}}^+ \propto V_g^2$  as has been suggested (ref. 2), then one may assume that the collector current for  $V_g < 150$  V is predominantly from gas phase ionization. Any estimate of x-ray limit from these data is likely to be a poor estimate. At  $V_g = 1000$ , however, the ion current is mostly due to x-ray photo emission and x-ray production estimates will be reasonably accurate.

Dushman (ref. 3) quotes an x-ray current equal to  $4 \times 10^{-8}$  A for a VG-1 A triode gauge operating at 10 mA and 1000 V. Figure 10 shows that the collector current of GG3 operating at 1000 V and 0.1 mA was  $6.7 \times 10^{-11}$  A of which  $\sim 6 \times 10^{-11}$  A can be identified as x-ray generated current. If we assume that  $I_{x\text{-ray}}^+ \propto I^-$  (emission), then  $I_{x\text{-ray}}^+$  (graphite) at 1000 V and 10 mA would be equal to  $6 \times 10^{-9}$  A. From this, we provisionally conclude that

$$\frac{\sigma_{x\text{-ray}}(\text{Tungsten})}{\sigma_{x\text{-ray}}(\text{Graphite})} = \frac{4 \times 10^{-8}}{6 \times 10^{-9}} = 6.6 .$$

If the x-ray production cross sections,  $\sigma$ , were proportional to atomic number, then we would expect

$$\frac{\sigma_{x\text{-ray}}(\text{Tungsten})}{\sigma_{x\text{-ray}}(\text{Graphite})} = \frac{Z_W}{Z_C} = 72/6 = 12 .$$

The principal problem with the first graphite gauge was the large outgassing load from the Grafoil components. Grafoil was chosen for the first prototype because of the ease of construction. Our outgassing data now show that the gas load could be reduced by a factor of 100 by substituting vitreous carbon. Gauge No. 4, shown in Figure 1, had vitreous carbon supports and vitreous carbon collector with pyrolytic graphite connectors. Outgassing would be further reduced if the gauge components were smaller. The size could have been reduced by a factor of two or three by using a vitreous carbon grid such as was later constructed. A possible way to make the graphite collector of very small size would be to evaporate a 500-1000 Å film of carbon on the inside of a glass tube. The size of the filament (electron source) could also be reduced by one-half, and still supply 0.1-3.0 mA with only about 18-24 W input. The size of supports and connectors could be reduced by almost an order of magnitude if the gauge were constructed in one step using phenolic resins to attach all parts.

### III. CONCLUSIONS AND RECOMMENDATIONS

Measurements of the adsorption and outgassing characteristics of four refractory carbons have confirmed that such materials are suitable for use in UHV, and that some are superior to refractory metals in some respects. The prototype gauges made from these materials, although quite simple and far from optimally designed, did show good sensitivity, durability, and apparently lower x-ray background. Thus, the feasibility of substituting carbon/graphite materials for refractory metals in UHV gauge construction has been proven.

Our results indicate that vitreous carbon is the best of the four forms of carbon tested for UHV use. Our results further suggest that a vitreous carbon Bayard-Alpert gauge would have approximately one order of magnitude lower x-ray background and nearly two orders of magnitude reduced memory effect. Similar improvements would be expected in the performance of mass-spectrometer ionization sources made from vitreous carbon.

If, as our prototype tests suggest, vitreous carbon components are significantly superior, then considerable improvements in the sensitivities and accuracy of much NASA instrumentation could be achieved simply and inexpensively by substituting vitreous carbon components for metal components. Many other areas of research and technology would also benefit from the immediate and inexpensive improvement of vacuum gauge and mass-spectrometer sensitivity and accuracy.

We therefore recommend that:

1. Vitreous carbon UHV components be further developed and tested in direct comparison with refractory metal components of like geometry;
2. A vitreous carbon Bayard-Alpert gauge be designed, constructed, and compared to a commercial, metal gauge; and
3. A vitreous carbon ionization chamber be fabricated and compared with the performance of a metal ionization chamber in a mass spectrometer.

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